5.3.2 SOLID-PHASE EXTRACTION BY CARBOPAK-B™ COLUMN

The Carbopak-B™ method currently (November 1998) is used for NWQL schedule 2051, which is for analysis of a broad spectrum of field-extracted pesticides.[9] Detailed descriptions of the method and the laboratory and field-extraction procedures can be found in Werner and others (1996). General equipment needs for solid-phase extraction are listed in table 5-7. For Carbopak-B™ SPE processing, obtain the SPE column, Carbopak-B™, 500 mg, precleaned; surrogate mixture and field-matrix spike-solution mixture for Carbopak-B™ SPE and PBW; ascorbic acid solution, 10 g/L; and reagent-grade sodium chloride (NaCl) 10 g/sample.

[9]The Carbopak-B™ method is graphitized carbon-based solid-phase extraction used with a high-performance liquid chromatographic analytical method for determining 41 pesticides and pesticide metabolites that are not readily amenable to gas chromatography or other high-temperature analytical techniques (Werner and others, 1996).

Quality-control samples are required as an integral part of the sampling program.

- Process a field blank with the first sample. Process additional field blanks about every 10 to 20 samples:
  - Use pesticide-grade blank water (PBW).
  - Process the blank in the same manner as the environmental water sample.
- Process field-matrix spikes about every 20 samples. When processing a field-matrix spike:
  - Use a 100-µL micropipet to add the field-matrix-spike solution to two of the triplicate samples. Follow the instructions provided in the spike kit.
  - Add the surrogate to every matrix-spiked sample and associated unspiked sample.
  - Record lot number and concentration of spike-solution mixture on the NWQL Schedule 2051 worksheet (fig. 5-3).

Before beginning field work, prepare an ascorbic acid solution in the office laboratory:

Each Carbopak-B™ requires 15 mL of ascorbic acid solution. Check that you have the volume needed before leaving for the field site(s).

The ascorbic acid solution must remain capped and chilled unless in use. The shelf life of the solution is 28 days—discard if shelf life has been exceeded or if the solution has been left uncapped or unchilled.

1. Place a tared, 1-L amber glass pesticide bottle (cleaned at the NWQL) on an analytical balance and fill to 500 g with PBW (pesticide-grade organic-free water purchased from NWQL DENSUPPL).
2. Empty a 5-g vial of ascorbic acid into the 500 g of PBW to obtain a 10-g/L ascorbic acid solution. Cap immediately and shake to dissolve.
3. Label the bottle with the date and preparer's name, contents of the solution, and the concentration of ascorbic acid.
4. Refrigerate the solution immediately and keep chilled until ready for field use. Transport to the field on ice in a foam sleeve.

Schedule 2051 Field Extraction Checklist and Reporting Sheet Solid-Phase Extraction, HPLC Analysis, Filtered Water

Station ID or Unique Number: _____
Station Name: _____
Date: _____
Time: _____
Collector: _____

❑ Filter Sample	0.7-µm glass fiber filter		
❑ SPE Cartridge Conditioning	Ascorbic acid solution [15 mL]	_____	mL
❑ Sample	Sample + bottle weight	_____	g
	– bottle tare weight	_____	g
	= sample weight	_____	g
❑ Surrogate	Solution lot number	_____	
	Volume added	_____	µL
– QA Samples Spike Mixture	Solution lot number	_____	
	Volume added	_____	µL
❑ Sample through Cartridge	Sample + plastic beaker	_____	g
	– plastic beaker	_____	g
	= volume of sample extracted	_____	mL
❑ Flow Rate	Start time	_____	hr:min
	Stop time	_____	hr:min

❑ Write Station ID Number and Sampling Date on Cartridge

❑ Remove Excess Water

❑ Replace Cartridge in Shipping Container and Store @ 4°C

❑ Comments:

Figure 5-3. Worksheet for Carbopak-B™ solid-phase extraction of pesticides.

Prepare to process samples onsite using the Carbopak-B™ column:

1. Put on disposable, powderless gloves during sample collection and processing. Cover a bench or table with a sheet of aluminum foil to make a clean work surface.
2. Collect and split samples using the procedures described in NFM 4 and NFM 5.1 (refer also Sandstrom and others, 1995; Werner and others, 1996).
3. Set up the equipment and assemble supplies on the clean work surface. Remove the aluminum foil wrapping from equipment.
4. Begin to fill out the NWQL Schedule 2051 worksheet (fig. 5-3), recording the type, lot number, and dry weight of the Carbopak-B™ SPE column.
5. Put on a new pair of gloves.
6. Tare the weight of a clean amber glass, 1-L sample bottle and a 1-L plastic beaker to the nearest gram using an analytical balance. Record the weight on the worksheet provided with each column.
7. Following the filtering instructions for general organic compounds (section 5.2.2.A or Sandstrom, 1995), filter about 1 L of sample through a glass microfiber filter into the tared bottle, leaving about 2 cm of headspace.
8. Weigh the filled bottle and record the weight on the worksheet (fig. 5-3).
9. Calculate and record the sample weight.

Extract the sample:

When extracting the sample, be sure to use the appropriate surrogate solution mixture supplied by the NWQL for the Carbopak-B™ SPE method. Add surrogate solution to all samples including field blanks, replicates, and field-matrix spikes.

Sample extraction must take place within 4 days of sample collection.

1. Withdraw surrogate mixture using a clean 100-µL micropipet and glass bore (detailed instructions on the use of a micropipet are included in the NWQL spike kit).
2. Insert the tip of the glass bore below the surface of the sample in the sample bottle and depress the plunger to deliver the surrogate mixture. (Tip the bottle, if necessary, to reach below the surface of the sample with the micropipet tip.) Keeping the plunger depressed, swirl sample with the pipetor several times and then withdraw the micropipet. Release plunger, then remove and discard the used glass bore.
3. Leave approximately 2 cm of headspace for the addition of NaCl.
4. Rinse the tip of the micropipet with methanol.
5. Add 10 g of NaCl to each sample. Cap and swirl the sample.
6. Process field-matrix spikes, if dictated by the study's quality-assurance plan. To process spikes, set aside three subsamples and spike two of the three subsamples with spike-solution mixture obtained from the NWQL spike kit. Follow the instructions provided with the kit.
7. Fill a clean glass graduated cylinder or beaker with 15 mL of ascorbic acid solution.
8. Using a metering pump fitted with 1/8-in. fluorocarbon polymer tubing and appropriate connectors:
   a. Turn on the pump.
      - Adjust the pump flow rate to deliver about 20 to 25 mL/min (1 drop per second).
      - Test the flow rate by pumping the cleaning solution into a graduated cylinder or beaker and timing with a stopwatch.
   b. Attach the outlet end of the pump tubing to the SPE-column adapter.
   c. Remove the SPE column from the shipping container and attach to the adapter. (The open end of the SPE column should fit tightly over the adapter; make sure the column is sealed completely against the lip of the adapter to create a leak-proof seal.)
   d. Place the inlet end of the pump tubing into 15 mL of ascorbic acid and pump the ascorbic acid solution through the column at a rate of 20 to 25 mL/min.
9. After all ascorbic acid solution has been pumped through the column, continue to pump air through the column for 1 minute. The conditioned column is now ready for sample extraction. **Extract sample onto the column within 8 hours of conditioning with ascorbic acid.**

10. Insert the inlet end of the pump's fluorocarbon polymer tubing into the sample bottle to begin sample extraction.

11. Pump sample through the Carbopak-B™ SPE column at a rate of 20 to 25 mL/min and collect extracted water in tared 1-L plastic beaker.

12. After the sample has been pumped through the column, turn off the pump and disconnect the SPE column.

13. Remove excess sample from the SPE column by using a syringe with 10 to 20 mL of air to push the excess sample into the tared, 1-L plastic beaker.

14. Weigh the beaker with the volume of sample processed through the SPE column (subtract tare weight of beaker from weight of beaker plus sample) and record the weight of the sample processed through the column on the worksheet (fig. 5-3).

15. Write the station identification number and the sampling date and time on the side of the SPE column and place the SPE column in a shipping container (40-mL glass or plastic ampoule). Complete the worksheet, wrap it around the shipping ampoule, and secure it with a rubber band or tape. Place SPE-column sample in a sealable bag. Keep a copy of the worksheet for the field folder.

16. Chill the SPE-column immediately and maintain at 4°C during storage and shipping.

Ship the SPE column to the laboratory immediately. Elution from the SPE column must be completed within 7 calendar days of extraction.

17. Field clean all equipment including the pump and tubing immediately after use (NFM 3) and before going to the next site. Rinse thoroughly with about 50 mL of a 0.2-percent solution of phosphate-free laboratory detergent, followed by about 50 mL of tap water or DIW to remove the detergent. Final rinse with 30 to 50 mL of methanol. Collect methanol rinse into an appropriate container. After cleaning, wrap all equipment apertures with aluminum foil.